

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

(12) UK Patent Application (19) GB (11) 2 183 626 (13) A

(43) Application published 10 Jun 1987

CG

(21) Application No 8628420

(22) Date of filing 27 Nov 1986

(30) Priority data

(31) 3542569

(32) 30 Nov 1985

(33) DE

(71) Applicant

Braas & Co. GmbH.

(Incorporated in FR Germany).

Frankfurter Landstrasse 2-4, D-6370 Oberursel, Federal Republic of Germany

(72) Inventors

Hans Erich Schulz,

Hans-Joachim Buhler,

Johann Dietz,

Manfred Weisweiler

(74) Agent and/or Address for Service

A. A. Thornton & Co., Northumberland House, 303-306 High Holborn, London WC1V 7LE

(51) INTCL⁴

B28C 7/00

(52) Domestic classification (Edition I)

C1H 120 420 620 718 730 739 812 814

U1S 1655 1699 C1H

(56) Documents cited

None

(58) Field of search

C1H

Selected US specifications from IPC sub-class C04B

(54) Coloured concrete roof tiles

(57) Concrete roof tiles are made by the extrusion press method from a mixture of Portland cement, sand, water and a coloured aggregate material (such as a solid or liquid iron oxide pigment or paint) having a colour corresponding to the desired finished appearance of the tile, in which

(a) the sand used either has a natural colour which corresponds to the desired finished appearance of the tile or is pre-treated (e.g. with iron oxide) so that it has the necessary colour,

(b) after the shaping step, but before the hot setting step, a setting retarder is applied uniformly to the visible surface of the tiles in an amount corresponding to the desired surface roughness, and

(c) after the hot setting step, the unset surface layer of the tiles is removed to form a rough surface.

GB 2 183 626 A

SPECIFICATION

Method of making coloured concrete roof tiles

5 This invention is concerned with a method of making coloured concrete roof tiles.

Concrete roof tiles are today produced virtually exclusively and throughout the world by the extrusion press method. The extrusion press method for producing concrete roof tiles is well known (see, for example, *Beton-stein-Zeitung*, 10/1959, pages 413 to 419) and does not have to be described here in detail. As a result of the method, a thin cement extrusion skin approximately 0.5 mm thick is formed on the surface of the tiles. The following surface refinement methods have become known for such concrete roof tiles, namely:

(a) The cement extrusion skin is left so that the concrete roof has a relatively smooth surface.

20 Experience has shown, however, that this untreated cement extrusion layer weathers away over a period of approximately 10 to 20 years. In this process, light-coloured grains of sand become visible on the tile surface and the surface becomes rough and a so-called pepper and salt structure results. The visual appearance of the tiles changes in the course of the weathering period. If some of the weathered concrete roof tiles on a roof area have to be replaced by new concrete roof tiles with a smooth surface, differences in the visual appearance of the tiles cannot be avoided and an undesirable impression is produced.

(b) Treatment of the concrete roof tile surface with acid after hot setting, preferably with 3 to 10% aqueous hydrochloric acid.

35 Although the treatment of concrete roof tiles with acid reduces the tendency to effloresce, an undesired change in the appearance of the tile surface in the course of the weathering period - exactly as described under (a) - cannot be avoided.

(c) A subsequent application of a slurry of cement or sand and cement to the concrete roof tile surface.

Cement slurries, including slurries containing super-fine sand aggregates, are known to form, as a rule, a glossy surface layer during the setting process. Over the weathering period, the tile surface increasingly acquires a more mat appearance. In addition, depending on its thickness, such a slurry weathers away in a certain period, for example, after about 10 years, throughout its entire thickness. The erosion process described above under (a) then sets in with a further alteration as mentioned in the appearance.

(d) A subsequent application of polymers or of a slurry comprising clear lacquer or paint or of glossy surface coatings and the like to the concrete roof tile surface.

The types of polymer used at present for coating concrete roof tile surfaces (acrylates, styrene acrylates, vinyl acrylates, etc.) have only limited durability to weathering. Observations have shown that such polymer coatings are weathered away completely after about 15 years. After complete erosion of the polymer coating, the weathering mechanism referred to under (a) and which is detrimental to the visual appearance then occurs.

(e) An additional application of coloured sand granulates to the slurry already applied to the concrete roof tile surface.

A coloured sand granulate applied to a cement slurry after the latter has been applied is also not resistant to weathering. As a result of limited adhesion to the base material, the coloured sand granulate is completely eroded in the course of time. The visual appearance of the tile surface alters simply as a result of the loss of granulates. The cement slurry becomes visible and after exposure, weathering as described above under (c) then occurs.

In short, the concrete roof tile surfaces obtainable with all these methods are insufficiently resistant to a change of appearance due to weathering.

We have now developed a method of forming a coloured surface on such concrete roof tiles which retains a substantially constant appearance when subjected to weathering.

85 According to the present invention, there is provided a method of making concrete roof tiles by the extrusion press method from a mixture of Portland cement, sand, water and a coloured aggregate material having a colour corresponding to the desired finished appearance of the tile, in which

(a) the sand used either has a natural colour which substantially corresponds to the desired finished appearance of the tile or, where such finished appearance is a red or reddish brown colour shade, contains up to about 1% by weight of iron oxide and has been subjected to a firing process so that its colouration substantially corresponds to such finished appearance.

(b) after the shaping step, but before the hot setting step, a setting retarder is applied uniformly to the visible surface of the tiles in an amount corresponding to the desired surface roughness, and

(c) after the hot setting step, the unset surface layer of the tiles is removed to form a rough surface.

105 In the initial mixture used in this method, it is preferred that the sand should have a particle size up to about 4 mm and it is also preferred that the mixture should contain from 3 to 3.8 parts by weight of sand and from 0.35 to 0.45 parts by weight of water per part by weight of Portland cement.

The combination of measures according to the invention, namely the colouration of the dry concrete mixture with a coloured aggregate material, the use of a coloured sand to produce the concrete mixture, and the application of a setting retarder to produce a definite surface roughness, ensures that the visual colour appearance of the tile surface does not change as a result of weathering period; i.e. it retains a constant appearance. With the method according to the invention, it is possible to obtain deliberately a particular surface roughness. The appearance of the tile surface may be varied from case to case, but is always controllable and the chosen appearance is retained after weathering. The graininess of the surface used in a particular case is visible at the tile surface in addition to the ground mass which determines the colour shade and because the sand grains protrude to a small extent from the bedding plane, the appearance of a naturally rough surface is obtained. The degree of roughness can be adjusted by the quantity of

120 125 130

setting retarder u ; after the hot setting step, the surface roughness becomes visible after removal of the unset material. The material removal is preferably effected by washing with water supplied under high pressure.

- Because the setting retarder acts to a relatively great depth which can, however, be adjusted by modifying agents, such as protein or cellulose additives; and can be limited, it is ensured that even after sand particles have been washed out or eroded as a consequence of weathering of the tile surface, neither the visual colour nor the visual structure, that is the rough appearance, is changed. In other words, even after a certain erosion of sand and parts of the coloured cement extrusion layer, no alteration will occur in the appearance of the tile surface once it is fixed because the same layer structure is always present.

- A method of producing concrete tiles with a surface simulating that of natural sandstone is described in German Offenlegungsschrift 2,812,496. In this method, a layer consisting of a mixture of concrete, quartz sand and paint which corresponds to the desired sandstone surface is first introduced into a mould. A further mixture of lower quality sand, chipings or gravel is applied to said layer. The entire mould is then bedded down and compacted by shaking on a vibrator. After removal and setting of the concrete tile slabs, the sandstone surface of the tile slabs is passed through a metal granule blasting device or sand blasting device in order to blast the surface of the tiles uniformly.

- This method does not relate to the production of concrete roof tiles and it does not make use of the extrusion press method; the use of sand of a particular colour is not mentioned. The essential difference is, however, that the roughening step using a sand blast only forms the uppermost layer of sand particules in a grain oriented manner. Because of its relatively low bond strength to the concrete layer lying thereunder, the sand particules of the uppermost layer are rapidly eroded when subjected to wear or weathering and the cement extrusion layer lying thereunder, along with the less or barely visible quartz particles, become visible.

The preferred manner of carrying out the method according to the invention is further described in detail below.

- Concrete roof tiles are made by the extrusion press method, the details of which are generally known, from a mixture of Portland cement, sand having a particle size of up to about 4 mm, and water in the ratio of 3 to 3.8 parts by weight of sand and 0.35 to 0.45 parts by weight of water per part by weight of cement. A coloured aggregate material is added to the mixture in the quantities usual for pigmentation, preferably up to 0.025 parts by weight of coloured aggregate material per part by weight of cement. Portland cement is used because it meets the established quality requirements and has the appropriate hydraulic setting properties. According to an important feature of the invention, a type of sand is used which, according to one alternative, corresponds in its natural colour to the desired visual colour appearance of the finished tile to the greatest

possible extent. This applies in particular for yellow, green, grey, brown and black colour shades. For green colour shades, for example, a sand of the rock type diabase green is used, for yellow colour shades yellow quartz or quartzite sand, for grey colour shades grey granite sand and for anthracite coloured and black colour shades a sand of the designation diabase grey or dark grey. For concrete roof tiles whose visual colour appearance is intended to have brown shades, a brown quartz sand may, for example, be used and similarly for certain reddish-brown colour shades for which, for example, reddish-brown quartz porphyry sand or reddish-brown granite sand may be used.

- According to another alternative, for concrete roof tiles whose desired visual colour appearance is a red shade or certain reddish-brown colour shades, a sand is used which contains up to about 1% of iron oxide. The desired red or reddish-brown colour shade is imparted to the sand by a firing process. For this purpose, the sand is fired in a suitable furnace, for example at a temperature of up to 600°C for 45 minutes or at a temperature of up to 500° for 60 minutes. In this process the firing time at the given maximum temperature determines the intensity of the red colouration. This means that, starting from its natural colour, the sand changes colour during the firing process with increasing firing time through reddish-brown shades right through to an intense dark red shade. At any given temperature no further appreciable increase in the red shade occurs after a certain firing time, that is a certain degree of saturation is reached. This period of time is specified for the particular furnace temperatures specified above as the maximum firing time. This means that the desired red or reddish-brown colour shade for the desired visual colour appearance of the finished tile can be exactly established by appropriate time and temperature control during the firing process.

- The coloured aggregate materials or colourants to be introduced into the concrete mixture can be colour-imparting pigments which are introduced into the mixture in solid form. These coloured aggregate substances may be solid iron oxide pigments which are colour-determining for the desired visual appearance of concrete roof tiles from yellow through green, brown, red to grey and black shades. However, in this case, the proportion of water in the concrete mixture must be controlled so as to ensure that the mixture is coloured throughout. Often it is more expedient and simpler to use iron oxide suspensions as colourants and to introduce them into the concrete mixture.

- After the concrete mixture has been produced from the above-mentioned Portland cement, sand, water and coloured aggregate material components, the tiles are produced by the shaping steps of the extrusion press method which is known per se. In order to obtain the naturally rough surface, after the shaping step, but before the hot setting step, a setting retarder is uniformly applied to the visible surface of the tiles. The quantity of setting retarder per unit area of surface is chosen to obtain the desired surface roughness. Hydroxy carboxylic acids, phosphates or lignin sulphonates may, for example, be used as the

setting retarder. The setting retarder is preferably used in a formulation together with a thickening agent, such as, protein or cellulose. The setting retarder may be sprayed on to the tile surface or may be applied by means of brushes. The liquid setting retarder formulation then diffuses more or less deeply into the roof tile. The depth of penetration is determined by the thickening component of the formulation, for example the protein or cellulose. The more thickener there is present in the formulation, the less the liquid setting retarder formulation penetrates the tile.

After application of the setting retarder, the tiles are subjected to the hot setting step. This is preferably carried out in a heated oven at approximately 60°C for a time of 6 to 8 hours.

After hot setting there is an unset surface layer on the tiles because of the setting retarder and the unset surface layer is then removed, preferably by washing with high pressure water.

After the washing step the concrete roof tiles are ready for use; they have a naturally rough surface which is determined by the grains of sand which are visible but only protrude minimally from the surface plane. The desired visual colour appearance of the tiles is produced by the coloured aggregate material and the colour of the sand introduced into the mixture.

When the tiles made by the method according to the invention are to be used in a climatic zone with unfavourable weathering conditions, it may be expedient to subject the tile surface additionally to a treatment with acid subsequent to the washing process and, if necessary, to carry out further surface sealing measures. Such an acid treatment preferably comprises spraying the tile surface with 3 to 5% aqueous hydrochloric acid, preferably followed by subsequent rinsing with water. Surface sealing subsequent thereto or coupled with the acid treatment may be carried out by applying suitable impregnating or coating materials. For example, final sealing of the tile surface may be carried out by spraying on an emulsified clear lacquer which may optionally be lightly filled with a filler, such as calcite, quartz, or talc.

Such an acid treatment serves to minimize or completely avoid concrete efflorescences. Surface sealing prevents weathering effects at least for a certain period of time. The impregnating or coating materials to be used are clear or transparent and do not affect either the visual colour appearance of the tiles or their natural rough surface structure as obtained by the method according to the invention.

55 CLAIMS

1. A method of making concrete roof tiles by the extrusion press method from a mixture of Portland cement, sand, water and a coloured aggregate material having a colour corresponding to the desired finished appearance of the tile, in which
 - (a) the sand used either has a natural colour which substantially corresponds to the desired finished appearance of the tile or, where such finished appearance is a red or reddish brown colour shade,

contains up to about 1% by weight of iron oxide and has been subjected to a firing process so that its colouration substantially corresponds to such finished appearance.

- (b) after the shaping step, but before the hot setting step, a setting retarder is applied uniformly to the visible surface of the tiles in an amount corresponding to the desired surface roughness, and
 - (c) after the hot setting step, the unset surface layer of the tiles is removed to form a rough surface.
2. A method according to claim 1, in which the unset surface layer is washed off with water supplied under high pressure.
3. A method according to claim 1 or 2, in which, subsequent to the removal of the unset surface layer, an acid treatment of tile surface is carried out.
4. A method according to claim 3, in which the acid treatment comprises spraying the tile with 3 to 5% aqueous hydrochloric acid and then rinsing the surface with water.
5. A method according to claim 3 or 4, in which, together with or subsequent to the acid treatment, the treated surface is sealed with an impregnating or coating material.
6. A method according to claim 5, in which sealing is effected by spraying an emulsified clear lacquer which may, if desired, contain a filler, on to the surface.
7. A method according to claim 6, in which the filler is calcite, quartz flour or talc.
8. A method according to any of claims 1 to 7, in which the setting retarder is a hydroxy carboxylic acid, a phosphate, or a lignin sulphonate.
9. A method according to claim 8, in which the setting retarder is used in a formulation which additionally contains a protein or cellulose as a thickening agent.
10. A method according to claim 8 or 9, in which from 6 to 14 g of liquid setting retarder formulation is sprayed or painted on to the tile surface.
11. A method according to any of claims 1 to 10, in which the coloured aggregate material is a solid iron oxide pigment or a liquid iron oxide paint which contains an iron oxide pigment.
12. A method according to claim 11, in which the liquid iron oxide paint is in an amount of 0.5 to 1.5% by weight, with respect to the weight of the cement.
13. A method according to any of claims 1 to 12, in which a natural sand is used which contains up to 1% by weight of iron oxide and the sand is fired at a temperature of up to 500°C for about 1 hour or at a temperature of up to 600°C for up to 45 minutes.